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METALLURGY OF RARE AND NOBLE METALS

Recovery of Nickel from Hydrate Pulps at Amino Carboxylic Cationites

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Abstract—Regularities of the sorption of nickel from hydrate pulps using amino carboxylic cationites ANKB-35, S 930, and TP 207 are investigated. Kinetic curves and sorption isotherms for these cationites are constructed. It is revealed that it is reasonable to absorb nickel from pulps at pH = 4–5. The desorption of nickel from cationites is investigated. It is shown that cationite TP 207 possesses the maximal sorption capacity.

Keywords: ion exchange, amino carboxylic cationite, hydroxide pulps

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Sorption technology is promising for recovering nickel from oxidized nickel ores [1]. This technology is based on sulfuric-acid leaching with the subsequent neutralization and sorption of nickel from hydrate pulp. It is shown in [2, 3] that amino carboxylic sorbents (ampholytes) are most suitable for the sorption of nickel from pulps. In this study we performed a comparative investigation of the sorption properties of ampholytes S 930 (Great Britain) and TP 207 (Germany), as well as the domestic ANKB-35 sorbent.

The ion exchangers used in this study refer to ampholytes and contain iminodiacetate groups in their structure [1, 4]:

$$R{-}CH_2{-}N\begin{matrix}\nearrow CH_2COOH\\ \searrow CH_2COOH\end{matrix}.$$

EXPERIMENTAL

In experiments, sorbents were preliminary treated with 20% sulfuric acid and washed with water to pH = 4–5. Their properties were investigated for a fraction of 0.63–0.81 mm.

Capacitive and kinetic characteristics of sorbents were determined for an actual production solution with the following composition: 1.2 Na, 1.6 Al, 6.0 Fe(III), 8.0 Mg, and 3 g/L H_2SO_4. For the experiments, the solution was neutralized to the required pH by lime milk. If necessary, the nickel content in the pulp was corrected by the introduction of seven-aqueous nickel sulfate of the chemically pure grade. Kinetic characteristics were recorded at the sorbent : solution volume ratio of 1 : 200.

The experiments on the regeneration of sorbents were performed in dynamic conditions in columns 30 mm in diameter with the sorbent layer 400 mm in height. The passing rate of the desorbing solution corresponded to two volumes per one sorbent volume for an hour. The solution flowing out of the column was collected in portions, and the nickel and sulfuric acid contents in them were evaluated.

Metal contents in experimental solutions were determined by the atomic-absorption method using a PerkinElmer spectrometer (United States), and the H_2SO_4 concentration was determined by the acidic–basic titration with a methyl-orange indicator.

Equilibrium sorption isotherms of nickel (the contacting time of the phases was 5 days) for all ampholytes have an abrupt convex character (Fig. 1), which indicates a large affinity of these sorbents to nickel and indicates to the possibility of its almost complete recovery from the liquid phase.

The maximal capacity of ampholytes is attained at the nickel concentration in the solution of about 3 g/L

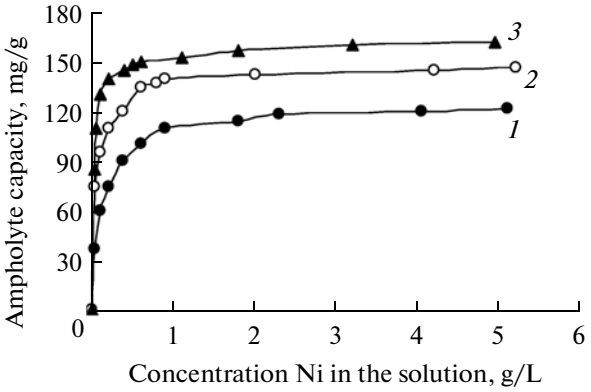

Fig. 1. Isotherms of sorption of nickel from the hydrate pulp on ampholytes (*1*) ANKB-35, (*2*) S 930, and (*3*) TP 207 at pH = 4.0.

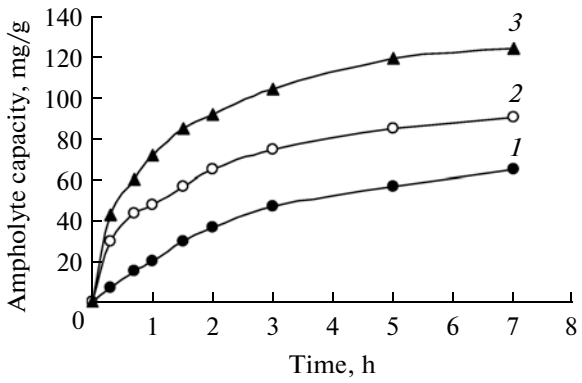

Fig. 2. Kinetics of sorption of nickel from the hydrate pulp on ampholytes (*1*) ANKB-35, (*2*) S 930, and (*3*) TP 207 at pH = 4.0.

and constitutes: 116 for ANKB-35, 150 for S 930, and 157 mg/g for TP 207 (or correspondingly, 35.8, 64.7, and 65.4 g/L).

It is seen from Fig. 2 that cationites S 930 and TP 207 possess the best kinetic characteristics. The absorption rate of nickel for the ANKB-35 domestic ampholyte is retarded, which will require increased volumes of equipment in industrial conditions. Allowing for this fact, its use for the sorption of nickel in industrial conditions should be recognized as inappropriate.

We further studied the capacities of ampholytes S 930 and TP 207 by the accompanying impurities, which are characteristic of solutions obtained during the sulfuric-acid leaching of oxidized nickel ores. The characteristics of sorption were analyzed in a range of pH = 1–7 (Fig. 3). We determined the affinities of elements Fe(III) > Ni > Al > Mg. This series agrees well with data on the investigations of chelate sorbents [5].

At pH of the onset of metal hydroxide formation, the ampholyte capacity is maximal. Then it abruptly decreases as hydroxide precipitates.

The capacity of sorbent TP 207 in regards to elements under study is higher than that of sorbent S 930 over the entire pH range under consideration. The maxima of capacity for TP 207 are shifted to a more acidic region, which is apparently explained by the higher content of iminodiacetate groups in it.

It is seen from Fig. 3 that it is most reasonable to perform the sorption of nickel from the pulp in a range of 4.0–4.5, which provides the maximal values of both the capacity for nickel and selectivity for impurities. It is evident that selectivity coefficients relative to impurity components, which are determined as the weight ratio of the capacity for the valuable component relative to the capacity for impurity, tend to infinity relative to aluminum and iron(III) in this pH range. In this case, the selectivity coefficient relative to magnesium for both types of ionites is rather low and constitutes only 1.7–3.0, which indicates the possible necessity of performing production operations for purifying the commercial strippant from magnesium impurities.

The regeneration of the sorbent involves operations of nickel desorption and washing acid from the sorbent. In order to determine the characteristics of regeneration, ampholytes TP 207 and S 930 were saturated with nickel from a model solution to capacities of 60.5 and 29.8 g/L, respectively.

Desorption with the subsequent washing of acid was performed according to the following flowchart. Sorbent saturated with nickel was separated from the pulp on a sieve; the sorbent was not washed during this operation. Then sorbents in an amount of 200 mL each were loaded into columns, after which 800 mL of the solution of sulfuric acid with the content of 200 g/L were passed through each column. The supply of the desorbing solution was performed upon reaching the ratio of the solution volume (V_{sol}) to the sorbent

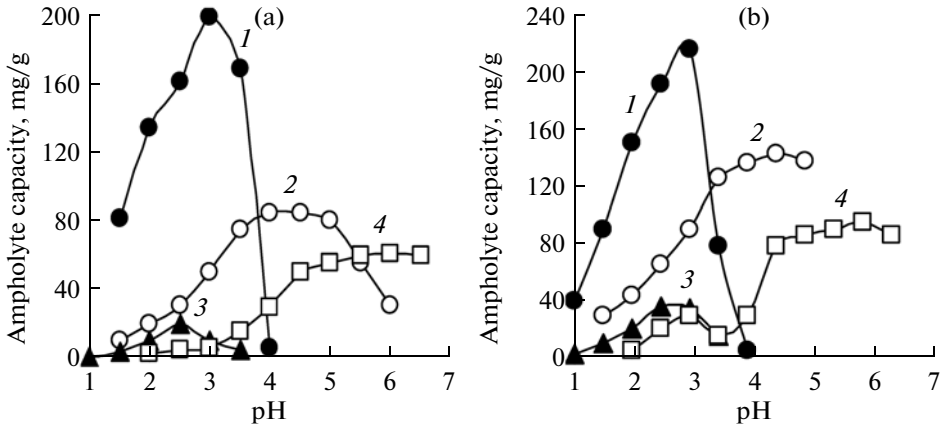

Fig. 3. Dependence of capacity of sorbents (a) S 930 and (b) TP 207 relative to ions (*1*) Fe^{3+}, (*2*) Ni^{2+}, (*3*) Al^{3+}, and (*4*) Mg^{2+}.

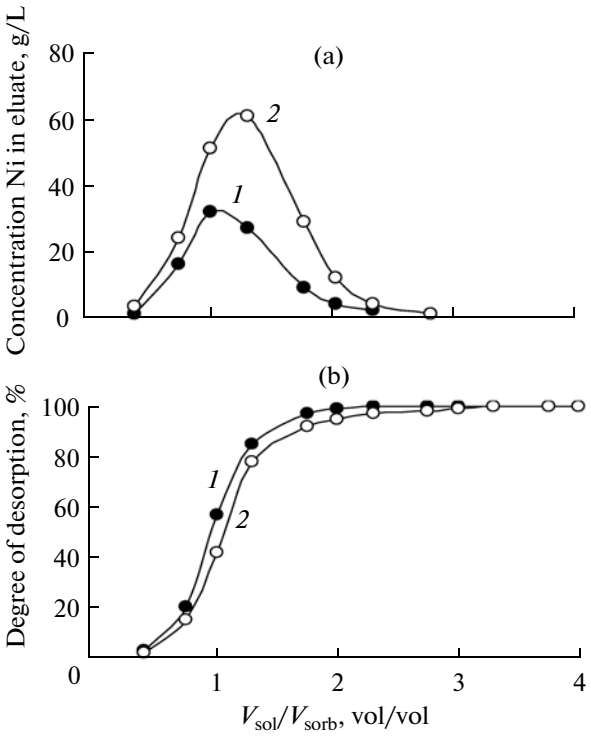

Fig. 4. (a) Differential and (b) integral curves of desorption of nickel from sorbents (*1*) S 930 and (*2*) TP 207.

volume (V_{sor}) of 4. Then acid was washed from the sorbent by passing water through the column.

We accepted the H_2SO_4 concentration in the desorbing solution as 200 g/L based on recommendations [4] in order to obtain the maximally high nickel contents in commercial stripping.

Differential and integral desorption curves of nickel depending on the volume of the passed solution per sorbent volume unit (Fig. 4) show that the degree of desorption of nickel weakly depends on the ampholyte grade and is determined by the volume of the passed sulfuric acid solution. It is seen from Fig. 4 that nickel is concentrated in 1.5 volumes of eluate by 90–95%.

When desorbing nickel, nickel ions in ampholytes are substituted by hydrogen ions, which is why the curve of increase in the sulfuric acid concentration in eluate fractions is shifted to the right of the curve of increase in the nickel concentration. Therefore, stripping fractions enriched by nickel have a low sulfuric acid content (Figs. 4, 5).

Sorbents are purified of acid (Fig. 5) when passing two volumes of water per one volume of sorbent by more than 95% practically regardless of ampholyte type.

Thus, both sorbents have good sorption and desorption characteristics, but due to the higher capacity of ampholyte TP 207, the nickel content in commercial fractions of eluate when using TP 207 in industrial conditions will increase by a factor of 1.5–1.7.

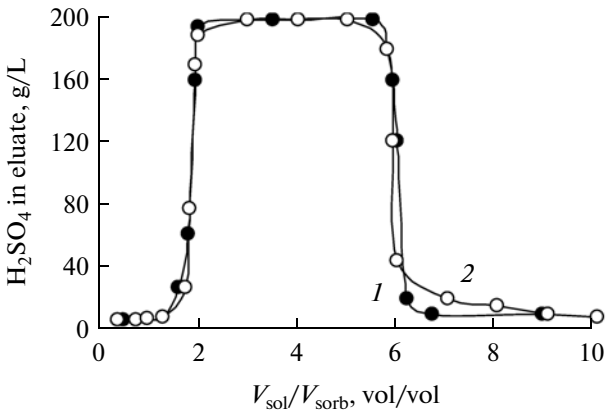

Fig. 5. Concentration of acid in the eluate with the sequential desorption of nickel and washing acid from sorbents (*1*) S 930 and (*2*) TP 207.

CONCLUSIONS

Sorption properties of a series of amino carboxylic cationites are investigated as applied to the sorption of nickel from hydrate pulps. It is shown that it is most reasonable to perform sorption at pH = 4.0–4.5, which ensures extremely high selectivity relative to aluminum and iron(III) impurities. In this case, selectivity relative to magnesium has a rather low level of values, which apparently will require the obligatory fulfillment of the additional saturation of the sorbent by part of the commercial stripping in production conditions in order to increase the purity of stripping.

The desorption of nickel is effectively performed by a solution with a content of sulfuric acid of 200 g/L with the acquisition of rich commercial stripping in the amount of 1.5–1.8 volumes per sorbent volume.

Thus, the results of experiments indicate the high efficiency of using ionites TP 207 and S 930 for the recovery of nickel from hydrate pulps of processing oxidized ores. Ampholyte TP 207 possesses higher capacity in regards to nickel (by a factor of 1.6), and its use is most preferable in industry.

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